THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Nickel-Molybdenum Thermocouple

R. D. POTTER: 'Calibration of a Nickel-Molybdenum Thermocouple.' *Jnl. Applied Physics*, 1954, vol. 25, Nov., pp. 1383–4.

During work on thermoelectric functions of alpha brass and aluminium, some difficulty has been experienced in spot-welding the usual types of thermocouple (Chromel-Alumel, iron-Constantan, copper-Constantan) to the specimens. It was found, however, that both nickel and molybdenum could be satisfactorily spot-welded to these materials. In view of a sparsity of information on calibration data for nickel-molybdenum thermocouples, the author made checks against a Chromel-Alumel standard couple. Results are reported in this paper, in table and curve form, showing temperature (°F.) *vs.* millivolts (abs.). The range covered is up to 2242°F. (50 mv.).

NICKEL

The Nickel Industry in 1953 and 1954

ORGANIZATION FOR EUROPEAN ECONOMIC CO-OPERATION: 'The Non-Ferrous Metal Industry in Europe.' *Published by O.E.E.C., Paris*, Jan., 1955; 154 pp.

The report is one of a series of studies on the general economic situation, which are carried out, for the principal industrial sectors, by the Technical Committees of the Organization. The metals covered are aluminium, copper, lead, zinc, nickel and tin, as produced from ores and concentrates.

A brief historical survey (from about 1950 onwards) introduces a review of the general situation obtaining in 1953 and the first half of 1954. This is followed by consideration of the main features of the market situation for each of the metals, a discussion of prices, the scope for investment in mining and other related development ventures, and a summary of prospects for the metals under review. Annexes to the report give statistical data on production and consumption in O.E.E.C. countries and their overseas territories, and tables showing the dependence of O.E.E.C. territories on imports.

The Committee has also incorporated in the publication a series of reports prepared by experts in the respective industries: the report on Nickel is by L. F. DENARO. This gives information on the following aspects of the subject: production (1934–1954, and estimate for 1960); production in Canada, New Caledonia, Japan, Cuba, U.S.A., South Africa, Greece, Finland, and other countries, with notes on reserves known to exist but not exploited; the main uses of nickel and the pattern of consumption in the O.E.E.C. countries; internal allocation procedure; the pattern of distribution in O.E.E.C. countries and in the free world; restrictions imposed in end-uses of nickel during the shortage of civilian supplies; prices, and the prospects for 1954.

The text of the report is amplified by numerous tables of statistical data.

Creep Characteristics of High-Purity Nickel

W. D. JENKINS, T. G. DIGGES and C. R. JOHNSON: 'Creep of High-Purity Nickel.' *Jnl. of Research, Nat. Bur. Standards*, 1954, vol. 53, Dec., pp. 329–52; R.P. 2551.

The tests reported in this comprehensive paper were made as one phase of a project which covers the study of the mechanism of creep in high-purity crystalline metals (nickel and copper) and comparison of the creep behaviour of these metals with that of binary nickel-copper alloys. The results of creep and other tests made at the National Bureau of Standards on high-purity copper have already been reported; relevant publications are listed in the bibliography attached to the present paper. Tensile tests previously made on high-purity nickel (*ibid.*, 1952, vol. 48, pp. 313–21), made over the range −320° to +1500°F. (−195° to +815°C.) had shown irregularities in some of the properties at about 80°–300°F. (27°–150°C.); recovery and recrystallization was found to predominate in specimens fractured at 1200° or 1500°F. (650° or 815°C.).

The material used for the creep tests reported in the present paper was from the same lot which had been used for the tensile tests: the analysis is given as follows (per cent. by weight):—nickel 99·85, copper 0·009, iron 0·04, manganese 0·03, silicon 0·11, carbon 0·007, sulphur 0·002, cobalt less than 0·01, oxygen 0·001, nitrogen 0·001, hydrogen 0·0002.

The tests were planned to determine the influence of stress, temperature, and prior-strain history, on the creep behaviour of initially annealed material; an evaluation was also made of the effect of pre-straining in creep (with or without subsequent annealing), on the hardness and tensile properties at room temperature. Metallographic examination was made of all specimens fractured in creep and of specimens broken in the room-temperature tensile tests after pre-straining in creep. The results are given

in great detail, in tabular and graphical form, and a large number of photomicrographs are shown. Some of the major observations recorded are summarized below:—

Discontinuous flow was observed in creep tests at 300°, 700°, 900°, and 1200°F. (150°, 370°, 480°, and 650°C.). The magnitude of the breaks in the creep-rate-extension curves varied with the temperature of test, and were affected by factors such as strain-ageing, strain-hardening, recovery, behaviour at the Curie point, recrystallization, and grain-growth.

The experimental values found for primary creep agreed fairly closely with results predicted by the theory of exhaustion and generation of dislocations as proposed by Mott and Nabarro and modified by other investigators.

Although, in general, the resistance of nickel to creep increased with lowering of test temperature, specially high resistance was found to occur at 300°F. (150°C.). This high resistance is attributed to a combination of strain-ageing and age-hardening and a relatively slow rate of recovery. Strain-ageing was especially prominent in the creep tests at that temperature. When the experimental values were plotted on log-log or semi-log (log of rate) bases non-linear relations between stress and secondary creep rate were usually obtained. It was also found that the second-stage creep rate was not a consistent index of the ductility of the nickel at the beginning of the third stage of creep, or at fracture.

Ductility at fracture was relatively high for all the creep conditions studied, except in one specimen. This specimen, tested at 1200°F. (650°C.) at a slow secondary-creep rate, showed general deterioration and brittleness at the time of fracture. In all the specimens tested at 300°, 700°, or 900°F. (150°, 370°, or 480°C.) internal disintegration was confined to the region of complete fracture, and fracture was preceded by appreciable necking. Cracks of microscopic dimensions were not a pre-requisite for initiation of the third stage of creep.

The yield and tensile strengths of specimens tested at room temperature after creep increased with the amount of pre-straining which had taken place at 900°F. (480°C.) and the increase was accompanied by fall in elongation values. Total reduction of area in the room-temperature tensile test of pre-strained specimens was, however, independent of the history of prior strain.

Rockwell B hardness, at room temperature, of specimens fractured in creep at 300°, 700°, or 900°F. (150°, 370°, or 480°C.) rose with increase in deformation in the neighbourhood of the complete fracture. This trend was observed also in a specimen which had been tested at a relatively high secondary rate of creep at 1200°F. (650°C.), but when slower rates had been used at the same temperature the effect was to lower the room-temperature hardness as the deformation increased in the vicinity of fracture.

Recrystallization occurred in specimens fractured in creep at 1200°F. (650°C.), and in some cases this change was associated with grain-growth. No evidence of recrystallization was observed in specimens which had been fractured in creep at the lower test temperatures. Fractures at 300°, 700°, and 900°F. (150°, 370°, and 480°C.) appeared to be predominantly transcrystalline; fractures at 1200°F. (650°C.) were intercrystalline.

The history of prior strain was found to exert a clearly marked effect on the rate of second-stage creep, and it is emphasized that the experimental results clearly demonstrate the importance of controlling rate of loading in creep testing.

Vapour Pressure of Nickel and Other Metals

A. B. SNIDER: 'Vapour-Pressure Curves for Carbon, Nickel, Iron, Chromium, Molybdenum, Tungsten, Copper, Tantalum and Aluminium.' *U.S. Atomic Energy Commission Publn.* CRD-T2B-171, 1952; declassified 1954.

Vapour pressures of the elements mentioned are tabulated over the pressure range 1 to 10^{-10} atm.

Nickel Carbonyl: Heat Capacity and Thermodynamics of Formation

J. E. SPICE, L. A. K. STAVELEY and G. A. HARROW: 'The Heat Capacity of Nickel Carbonyl and the Thermodynamics of its Formation from Nickel and Carbon Monoxide.' *Jnl. Chemical Soc.*, 1955, Jan., pp. 100-4.

Values are given for the heat of fusion of nickel carbonyl, for its melting point, and for its heat capacity between 90° and 270°K. (−183° and −3°C.). The thermodynamics of the reaction $Ni+4CO \rightleftharpoons Ni(CO)_4(g)$ are discussed.

Chemical Analysis of Steel: Determination of Nickel

See abstract on p. 47.

Determination of Nickel and Other Elements in High-Temperature Alloys

See abstract on p. 55.

ELECTRODEPOSITION AND OTHER COATING METHODS

Nickel Plating of Wire

E. C. SLICK: 'Composite Wire by Electroplating.' *Wire and Wire Products*, 1954, vol. 29, Nov., pp. 1342-6, 1362-8.

After introductory notes on methods generally adopted for the cleaning of wire which is to be electroplated in a continuous plant, the technique of electrodeposition is described by reference to nickel-plating of steel wire. Mechanical operations which will be carried out on the wire necessitate ductility of the coating, and it has been found that a fluoborate solution of pH 2·7-3·5 and operated at 130°F. (55°C.) gives satisfactory results. The purity of the solution is an important factor: excessive amounts of copper or of iron tend to cause discolouration of

the plating, brittleness, high internal stress, and pitting. Periodic or continuous filtration should be ensured. A further factor of importance in production of uniform coatings on wire is the shape of the anodes. The wire progressing through the tank in helicoidal form calls for anodes which conform to a circular contour, in order to maintain uniform current distribution and thickness of coating. Recommendations on form and placing of anodes are made in the paper. It is essential to use anodes of high purity and it is desirable that they should be bagged: cotton and similar material has been found unsatisfactory, due to attack by the fluoborate solution, but bags made from synthetic fibres such as Vinyon have been successfully used.

The condition of the steel wire is also significant: it should be uniform in size within any one coil, and, where possible, from one coil to another, since the coils are welded together for continuous plating: it should be of $\frac{1}{4}$- to $\frac{1}{2}$-hard temper, and have a thoroughly clean and smooth surface. Pickling should be carefully controlled, since although it is desirable to etch the surface of the wire to promote a good mechanical bond, such treatment comprises only a very fine micro-etch, and does not produce the excessive roughness caused by over-pickling.

With regard to drawing lubricants for use on plated wires, the writer of this article points out that selection is contingent on the type of wire being drawn and the conditions of operation. For heavy-section nickel-plated wire dry soaps may be used; for copper-plated materials wet methods are to be preferred. An initial pass through tallow is recommended. With plated wire of intermediate sizes (0·040–0·005 in. diameter) fluid-type compounds may be employed. Per cent. reduction varies somewhat with the material being drawn: nickel-plated steel can be reduced by 30 per cent. in each pass, but for nickel-plated copper reductions of 15 per cent. give better results. Very fine wire (0·005 in. or below) is drawn at much higher speeds than the thicker gauges, and a more dilute lubricant should be used.

There is still debate in the wire industry with regard to the most efficient shape of die, and much depends on the type of lubricant used, the percentage reduction per pass, and other variants. Entrance angle, bearing length and blended radii must be controlled to minimize the power required, to reduce friction and wear, to keep the temperature down, to maintain high drawing speeds, to obtain good die life, and to ensure a satisfactory surface finish on the product.

Final annealing treatment varies according to the nature of the plated wire product; the temperature of this treatment should usually be the lowest one which will restore ductility to the higher-melting-point metal involved; for nickel-plated low-carbon steel wire the range is about 1350°–1400°F. (730°–760°C.).

Uses of plated wire include components used in power transmission, jewellery, kitchen utensils, racks, screens, and other items embodying wire components.

Electrodeposition of Metals by the Dalic Brush-Plating Process

M. RUBINSTEIN: 'Brush Plating Now Practical.' *Materials and Methods*, 1954, vol. 40, Dec., pp. 98–101.

In the Dalic brush-plating process, developed in France, a cotton swab, saturated with an electrolytic solution, is made anodic and is run to and fro over the (cathode) surface to be plated. It is claimed that even on materials such as aluminium and stainless steel the abrasion of the anode pad against the surface of the basis material results in a high degree of adhesion, without any special preparation or pre-etching. Brush plating is also stated to produce coatings which are pore-free and practically immune from hydrogen embrittlement. At the present stage of development the plating so deposited is bright only in films of 0·0005-in. thickness or below: in thicknesses normally used the deposits have a dull finish, and require polishing. A table in this article lists the limitations in thickness obtainable in Dalic coatings of some twenty metals, and notes the characteristics of the respective coatings, including nickel, chromium, cobalt, copper, iron, silver, platinum and rhodium.

It is claimed that the new solutions developed for use in this process are the main clue to the improvements which it has been possible to effect in brush plating. The baths are highly concentrated, are designed to plate at high current densities (300–6000 amp. per sq. ft. of contact area), do not oxidize the surface of the basis metal, and are non-toxic.

To provide for plating of objects of varying shape and size, three types of stylus or brush are now used:—the cap, plug, and roller forms: these are illustrated, and their uses are described.

The many applications for which the Dalic process is considered to be useful are briefly outlined: they include building-up and repair work, surfacing to reduce friction, coating of electrical contacts, electronic equipment, and other small parts, plating of strip and wire, and the formation of plastic-metal combinations.

Metallurgical Hardness and Buffing Quality of Electrodeposited Coatings

H. FAINT, and R. S. MODJESKA: 'A Correlation of Metallurgical Hardness and Buffability.' *Plating*, 1954, vol. 41, Dec., pp. 1422–5.

Full form of paper referred to, from abstract source, in *Nickel Bulletin*, 1954, vol. 27, Nos. 7–8, p. 129.

Electroless Nickel Plating: Commercial Experience

G. GUTZEIT and R. W. LANDON: 'A Large-Scale Electroless Nickel Custom Plating Shop.' *Plating*, 1954, vol. 41, Dec., pp. 1416–20, disc., pp. 1420–1.

In this paper, presented at the 41st Annual Convention of the American Electroplaters' Society, the authors briefly discuss the basic principles of the Kanigen electroless process and some of the factors essential for its successful operation. These notes embody information broadly similar to that which has been given in recent articles on this subject.

The major portion of the paper consists in the description of the plant and plating methods used by General American Transportation Corporation, by whom the process has been commercially developed.

Electroless Nickel: Critical Appreciation of its Potential Value

N. HALL: 'Electroplated vs. Electroless Nickel.' *Metal Finishing*, 1955, vol. 53, Feb., p. 51.

Editorial, very briefly summarizing the major advantages and limitations of electroless plating, so far as they can be assessed at the present stage of development. For a fuller consideration of the subject, and discussion of the position of electroless plating vis-à-vis electrodeposited nickel, attention may be directed to the review by BRENNER (*ibid.*, 1954, vol. 52, Nov. and Dec.: *Nickel Bulletin*, 1955, vol. 28, No. 2, pp. 28–9), and to other items of literature recently reviewed in *Nickel Bulletin*, e.g., 1954, vol. 27, No. 9, pp. 166–7.

NON-FERROUS ALLOYS

Fabrication of Nickel Alloys

'Twenty-Five Years of Nickel-Alloy Fabrication. *Metallurgia*, 1954, vol. 50, Dec., pp. 257–62.

This article draws attention to the silver jubilee of the Zenith Works of Messrs. Henry Wiggin and Company, Limited, originally opened in 1929 as a mill designed for the production of tubes of small diameter and of specialized types. Many important contributions have been made by this works to the development of methods for fabrication of nickel alloys: one of the most outstanding of these is the application of extrusion methods, first in tube manufacture, and later in production of gas-turbine blading and other forms. The early history of the works, its wartime activities, and its participation in development of aircraft gas-turbine components in the Nimonic alloys are reviewed. Equipment and current methods of operation are described and illustrated.

The Nickel-Chromium-NiAl System

I. I. KORNILOV and R. S. MINTS: 'Melting Diagram of the System Nickel-Chromium-NiAl.' *Doklady Akad. Nauk S.S.S.R.*, 1954, vol. 94, pp. 1085–8.

A summary of the results is given in *Metallurgical Abstracts*, 1954, vol. 22, Dec., p. 303.

Magnetostriction in Nickel-Copper Alloys at Sub-Zero Temperatures

Y. SHIRAKAWA and K. NUMAKURA: 'On the Longitudinal Magnetostriction of Nickel–Copper Alloys at Low Temperatures.' *Nippon Kinzoku Gakkai-Si*, 1954, vol. 18, No. 2, pp. 73–7.

Measurement of magnetostriction in nickel and nine nickel-copper alloys (containing 4·76–42·93 per cent. of copper) in longitudinal magnetic fields up to 1600 Oersted, at 0°, −95° and −195°C. For summary of results, see *Metallurgical Abstracts*, 1954, vol. 22, Nov., p. 183.

Superconducting Nickel-Bismuth Alloys

G. S. ZHDANOV ET AL: 'The Structure of Superconductors. I. Study of the System Bismuth-Nickel. Preparation and Investigation of Single Crystals of Bi_3Ni.' *Zhurnal Eksper. Teor. Fiz.*, 1953, vol. 25, No. 1, pp. 115–22; *Physics Abstracts*, 1954, vol. 57, Dec., Abs. 11,530.

'Diagrams showing the hardness and density of solid phases in the bismuth-nickel system were constructed. BiNi, which has the NiAs structure, was both the hardest and densest phase found. Either BiNi or Bi_3Ni crystals could be produced from the same melt, depending on the degree of superheating. A technique for production of the Bi_3Ni crystals was developed, and the unit cell was measured.'

See also—

V. P. GLAGOLEVA and G. S. ZHDANOV: 'The Structures of Superconductors. VII. X-Ray Crystallographic Determination of the Structure Bi_3Ni.' *Zhurnal Eksper. Teor. Fiz.*, 1954, vol. 26, No. 3, pp. 337–44; *Physics Abstracts*, 1954, vol. 57, Dec., Abs. 11,567.

'The unit cell of Bi_3Ni is orthorhombic, with the following dimensions:—

$$a = 8{\cdot}875 \pm 0{\cdot}006 \text{ A}$$
$$b = 4{\cdot}112 \pm 0{\cdot}004 \text{ A}$$
$$c = 11{\cdot}477 \pm 0{\cdot}008 \text{ A.}'$$

Palladium-Nickel-Chromium Alloy for Brazing High-Temperature Alloys

See abstract on p. 51.

Oxidation of Nickel-Molybdenum Alloys

See abstract on p. 49.

Corrosion of Condenser Tubes in contact with Polluted Waters

See abstract on p. 53.

NICKEL-IRON ALLOYS

Casting of Permanent Magnets

A. J. J. KOCH and K. J. DE VOS: 'Some Methods for Casting Anisotropic Permanent Magnet Alloys of Iron–Cobalt–Nickel–Aluminium Base.' *21st International Foundry Congress, Florence*, Sept., 1954, Paper No. 48; 7 pp.

This paper reports a comparison of methods of casting permanent magnets from alloys of the following nominal composition: cobalt 24, nickel 14, aluminium 8, copper 3, iron 50, per cent. After a consideration of the general properties of this type of material, and of the requirements which must be satisfied by a production method which can be used on a commercial scale, the authors discuss results

attainable by various methods of casting. The information is based on work done in the laboratory and works of N.V. Philips Gloeilampenfabrieken, Eindhoven. The relative advantages and limitations of the following methods of casting are considered:—

(i) Casting in Green Sand Moulds (of natural or synthetic sands).
(ii) Casting in Permanent Moulds, including centrifugal casting.
(iii) Shell Moulding (use of resin-bonded moulds).
(iv) Casting in Exothermic Moulding Materials.
(v) Casting by the Lost-Wax Method.

The results of the experiments lead to the conclusion that the use of the shell moulding (C) process permits wider latitude in selection of the composition of the base moulding sand than is permissible with other casting processes, and that such advantage might well be exploited in relation to this technique. The rate of cooling of the casting in the mould is dependent, among other factors, on the material of the mould. Cooling in resin-bonded moulds (as used in the C process) is, in general, somewhat slower than in green-sand moulds. Among the green sands, cooling rate is higher when zircon or olivine sands are used than with silica sand. Casting methods are discussed also in relation to production of controlled crystal orientation.

Permanent-Magnet Materials having Specially High Coercive Force

T. O. PAINE and L. I. MENDELSOHN: 'High-Coercive-Force Permanent-Magnet Materials and their Applications.' *Electrical Engineering*, 1954, vol. 76, Oct., pp. 891–5.
Reprint of paper presented before American Institute of Electrical Engineers, January, 1954.

The paper is concerned with permanent-magnet materials characterized by intrinsic coercive force (H_{ci}) greater than 750 oersteds: designs offering particularly advantageous application for such materials are indicated, and some problems arising in their use are considered.

In a table and figures the authors review the magnetic properties of ten permanent-magnet materials falling into this group:—

Silmanal, a ductile Heusler alloy, of which a typical composition is silver 86·75, manganese 8·8, aluminium 4·45 per cent.

Cobalt–platinum order–hardening alloys of the 77–23 per cent. type. (Reference is also directed to iron-platinum permanent-magnet alloys.)

Manganese–bismuth alloy, composed of the intermetallic compound MnBi (bismuth 79, manganese 21 per cent.).

Barium–iron oxide, a hexagonal oxide compound having the formula $BaO.6Fe_2O_3$, which has been given the name Ferroxdure. (Directional and non-directional types of magnet are discussed.)

Vectolite, a cobalt–iron oxide containing Co_2O_3 (26%) Fe_2O_3 (30%) and Fe_2O_4 (44%).

Cunife, a ductile and machinable alloy containing copper 60, nickel 20, iron 20 per cent., which requires heavy cold-working before ageing, in order to develop optimum magnetic properties.

Alnico VII, one of the precipitation-hardening series based on aluminium, nickel, cobalt and iron: a typical composition is given as aluminium 8, nickel 18, cobalt 24, titanium 5, copper 3, per cent., balance iron. The alloy has the highest coercive force of any of this group of alloys.

Cunico, an alloy of type closely similar to Cunife; it contains copper 50, nickel 21, cobalt 29, per cent.; prior to ageing it is ductile and machinable.

Alnico VI, which has both coercive force and induction intermediate between those of Alnico V and Alnico VII. The composition of No. VI is given as aluminium 8, nickel 15, cobalt 24, copper 3, titanium 1, per cent., balance iron.

Properties typical of the above materials are shown in the table below:

The second part of this paper comprises a review of the opportunities which are open to magnets of the above materials, in view of their specially high coercive force, combined with the other properties described. The advantages discussed are:—

(1) the possibility of making smaller, more compact and highly efficient magnets;
(2) the value of high coercive force in applications for which the magnets must be magnetized before assembly into the final circuit;
(3) resistance of such magnets to external demagnetizing fields;

Material	Hci	Hc	Br	$(BH)m \times 10^{-6}$
1 Silmanal	6,000	550	550	0·076
2 Cobalt–platinum	5,000	4,000	5,700	7·5
3 Manganese–bismuth	—	3,400	4,300	4·3
4 Directional barium–iron oxide	—	2,000	3,500	3·0
5 Non–directional barium–iron oxide ..	2,900	1,500	2,000	0·8
6 Vectolite	1,100	1,000	1,600	0·6
7 Cunife VII	1,100	1,000	2,800	1·2
8 Alnico VII	1,050	1,000	7,200	2·75
9 Cunico IV	900	800	2,900	0·8
10 Alnico VI	775	750	10,000	3·5

(4) suitability of the higher-coercive-force magnets for use in repulsion systems;
(5) suitability for magnets of short length, e.g., in cases in which the ratio of the length of the magnet to its diameter is in the region of one.

Typical examples are given of applications in which these advantages are demonstrated.

Determination of Nickel and Other Elements in Nickel-Iron Alloys

See abstract on p. 55.

Properties of Nickel-containing 'Ferrites'

H. P. J. WIJN, E. W. GORTER, C. J. ESVELDT and P. GELDERMANS: 'Conditions for Square Hysteresis Loops in Ferrites.' *Philips Technical Rev.*, 1954, vol. 16, Aug., pp. 49–58.

The article deals with the fundamental conditions requisite for obtaining 'ferrites' characterized by rectangular hysteresis loops. The composition and properties of some materials fulfilling the requirements are discussed: these include nickel-containing 'ferrites'. The applications open to such materials are also considered.

CONSTRUCTIONAL STEELS

Nickel-Alloy Steels for Machine Tools

MOND NICKEL CO., LTD.: 'Nickel-Alloy Steels for Machine Tools.' *Publn.* 233,* 1955, 24 pp.

This publication briefly reviews the qualities called for in machine tools, and gives detailed consideration to the use of nickel-alloy steels, as providing such qualities. The properties obtainable in case-hardening and direct-hardening grades of these steels are shown in a comprehensive series of tables and charts, recommendations are made on heat-treatment, and the suitability of the respective steels for specific applications in machine tools is indicated.

Tensile and Compression Tests on High-Temperature Sheet Materials

See abstract on p. 48.

High-Temperature Properties of Steels used in Power Stations

See abstract on p. 48.

Determination of Titanium in Steel

See abstract on p. 55.

Chemical Analysis of Steel: German Practice

R. WEIHRICH: 'Chemical Analysis in the Steel Industry.' Published by *F. Enke Verlag, Stuttgart*, 1954; viii + 223 pp.

The major proportion of this book is concerned with sampling and analysis of steels, ferro-alloys, ores and slags: the remainder (about one quarter) covers potentiometric, colorimetric, photometric and polarographic procedures for estimation of individual elements, and contains notes on applications of micro- and spectrographic analysis. Some tables of data relevant to chemical analysis, and a *bibliography* of major German textbook literature on chemical analysis are also included. The information may be considered to be representative of procedures currently used in Germany. Determination of nickel is dealt with on pp. 47–52, 170, 182 and 198.

* *We shall be pleased to supply a free copy of this publication.*

HEAT- AND CORROSION-RESISTING MATERIALS

Vacuum Melting of High-Nickel Alloys

R. K. MCKECHNIE, D. W. GREEN and W. F. MOORE: 'Vacuum Melting Improves Alloys Properties and Workability.' *Jnl. of Metals*, 1954, vol. 6, Dec., pp. 1364–7.

This paper is based on experience in the plant of General Electric Company, Schenectady, where vacuum melting is being successfully applied to production of iron-, cobalt- and nickel-base alloys. Operating technique is described in detail as exemplified by methods employed in the manufacture of the nickel-base alloy M. 252, which has the following nominal percentage composition: iron 5 max., chromium 19, cobalt 10, molybdenum 10, titanium 2·5, aluminium 1·0, carbon 0·15, manganese 0·75, silicon 0·50, nickel balance. Typical gas contents of vacuum-melted M. 252 are oxygen 0·0005–0·001, nitrogen 0·002–0·003 per cent. The improved workability obtained in the clean, 'gas-free' material is demonstrated by an account of the high yields obtained in routine production, and by reference to the relative ease with which the alloy can be forged in manufacture of gas-turbine blades.

Results obtainable by vacuum melting are then discussed in two categories: (1) improvements in materials which can also be processed by conventional methods, and (2) production of materials for which, due to their composition, vacuum melting is essential. In case (1) the improvements can be ascribed to the use of the vacuum melting technique *per se*, in case (2) the advantage is that by vacuum melting it is possible to produce new alloys, e.g., an M. 252 composition without manganese or silicon, which could not be made by other means.

As an example of the first category, the authors show a comparison of the properties, in the range 1200°-1500°F. (650°-815°C.), of conventionally melted, vacuum-melted normal, and vacuum-melted manganese-, and silicon-free M. 252. It is demonstrated that vacuum melting alone produces an alloy significantly more ductile than the similar alloy made by normal melting methods, and that the alloy from which manganese and silicon are absent gives, at 1350°F. (735°C.) an elongation value almost three times that of the vacuum-melted alloy in which these elements are present. A further example of improved properties is given by data on the cobalt-base alloy 1570 (iron 2·5, chromium 20·0, nickel 29·0, tungsten 7·0, titanium 4·2, carbon 0·20, per cent., balance cobalt).

Specific Heat of Nickel-Chromium Alloys at High Temperatures

H. MASUMOTO, M. SUGIHARA and M. TAKAHASHI: 'On an Anomaly in Specific Heat at High Temperatures in α-Phase Alloys of Nickel and Chromium.' *Nippon Kinzoku Gakkai-Si*, 1954, vol. 18, No. 2, pp. 85–7.

Report of study of the specific heats of twelve nickel-chromium alloys containing up to 49·32 per cent. chromium. The temperature range covered was 100°–800°C. For results see *Metallurgical Abstracts*, 1954, vol. 22, Nov., p. 183.

Cutting Fluids for Use in Machining Nimonic Alloys

A. E. LAWSON: 'Cutting Fluids. Characteristics of Compounds for Use in the Machining of Nimonic Alloys.' *Aircraft Production*, 1955, vol. 17, Jan., pp. 38–9.

The author outlines the machining characteristics of nickel-chromium-base alloys, and reports conclusions (derived from service experience) with regard to the optimum types of cutting fluid for use in turning and broaching, tapping and drilling, and milling and grinding of the Nimonic series of alloys, which are widely used for aircraft gas-turbine blading and other gas-turbine components.

Tensile and Compression Tests on High-Temperature Sheet Materials

P. J. HUGHES, J. E. INGE and S. B. PROSSER: 'Tensile and Compressive Stress-Strain Properties of Some High-Strength Sheet Alloys.' *Nat. Advisory Committee for Aeronautics, Tech. Note* 3315, Nov., 1954; 32 pp.

Missiles and aircraft operating at supersonic speeds must be constructed in materials which can withstand the adverse effects of elevated temperatures. Researches are in progress, under the auspices of the National Advisory Committee for Aeronautics and other organisations, to provide information on the properties of various ferrous and non-ferrous materials which merit consideration for such applications. This report covers investigations on sheet material of the types shown in the table below.

Conventional short-time tests were made, with the specimens exposed to the test temperature for ½ hour prior to loading. Tensile and compressive stress-strain data, taken in the direction of rolling and in the transverse direction, are given for all these materials at temperatures up to 1000°F. (540°C.), and additional compressive values are quoted for Stainless W and for Inconel at 1200°F. (650°C.). Information on the treatments to which the respective materials were subjected before test are tabulated; they were designed to provide ultimate tensile strengths, at room temperature, in the 170,000–220,000 p.s.i. (76–100 tons per sq. in.) range.

The properties of any one of the materials were found to be essentially the same in both directions; at any given temperature the tensile curves were slightly lower than the corresponding compressive curves. With Inconel X there was little effect of temperature on tensile and compressive yield stresses, and a reduction of only about 35 per cent. in ultimate tensile stress up to 1200°F. (650°C.), as compared with a large fall in strength for the other materials at temperatures above 800°F. (425°C.).

High-Temperature Properties of Steels used in Power Stations

COMMISSION D'ÉTUDE DES MÉTAUX À CHAUD DE L'IRSID (INSTITUT DE RECHERCHES DE LA SIDÉRURGIE): 'High-Temperature Properties of Chromium-Molybdenum and Austenitic Chromium-Nickel Steels.' *Rev. de Métallurgie*, 1954, vol. 51, Dec., pp. 845–68.

This report comprises the results of three series of tests made on steels in use in French (and other) power stations. In all cases tests were made at temperatures within the range 500°–700°C. (approx.), i.e., at temperatures at which such steels are normally used. The scope of the individual tests is indicated below.

I. *Steel containing chromium* 0·6, *molybdenum* 0·6, *per cent.* (to AFNOR specification 15 CD 2),

as used for boiler and superheater tubes, pipe stills in the oil industry, etc. Tested at 500°C.

II. *Steel containing chromium* 2·25, *molybdenum* 1 *per cent.* (to AFNOR specification 10 CD 9),

as used for piping of steam plant and in the oil industry. Tested at 535° and 550°C.

III. *Steel containing chromium* 22, *nickel* 18 *per cent.* Properties at 600° and 700°C. were determined, and a study was made of structural changes occurring under prolonged exposure in this range of temperature, with particular reference to the development of sigma phase.

For all three steels details are given of room-temperature properties, short- and long-time properties

Materials	C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %	Ti %	Nb %	Al %	Fe %
S.A.E. 4340	0·42	0·24	0·78	0·027	0·018	1·79	0·80	0·33	—	—	—	Bal.
Hy-Tuf	0·25	1·5	1·30	—	—	1·80	—	0·40	—	—	—	Bal.
Stainless W	0·05	0·57	0·54	0·006	0·01	6·73	16·80	—	0·58	—	—	Bal.
Inconel X	0·04	0·40	0·50	—	—	73·0	15·0	—	2·5	1·0	0·7	7·0

within the ranges of elevated temperatures indicated, and the structures associated with the various conditions. In the austenitic steel, clear indications of sigma formation were found, with associated deterioration of ductility.

Oxidation of Nickel-Molybdenum Alloys

S. S. BRENNER: 'Oxidation of Iron-Molybdenum and Nickel-Molybdenum Alloys.' *Jnl. Electrochemical Soc.*, 1955, vol. 102, Jan., pp. 7–15.

The phenomenon of 'catastrophic oxidation' occurring in molybdenum-containing materials is now a well-recognized problem of high-temperature engineering. Although there is at present some divergence of opinion with regard to the exact mechanism of the oxidation effect, it is generally agreed that the trouble is due mainly to the presence of molybdic oxide (MoO_3). The low melting point of this oxide (795°C.), and its high vapour pressure (0·25 atm. at 1000°C.), prevent the formation of protective oxide which would normally be formed in the absence of MoO_3.

Most of the data published up till now have been derived from experiments on commercial alloys, but the present paper deals with the behaviour of laboratory-prepared binary iron-molybdenum and nickel-molybdenum alloys, and the alloys referred to in the following abstract were also of specially prepared types.

The binary iron alloys contained up to 12·5 at. per cent. of molybdenum, the nickel-base alloys ranged up to 19·7 at. per cent. molybdenum. On both types tests were made, on thin rolled strip, in still air and in flowing oxygen atmospheres, over the temperature range 800°–1000°C.

From the considerable experimental data recorded, the author concludes that molybdenum significantly alters the high-temperature oxide structure normally found on iron or on nickel. Due to the low solubility of molybdenum in NiO and in the iron oxides, molybdenum-rich oxide layers of MoO_2 and molybdate are formed. The volatile MoO_3 is, however, prevented from forming, and no catastrophic oxidation occurs within the range of temperature studied.

Molybdenum considerably reduces the oxidation rate of iron. Molybdenum-enrichment at the alloy surface during the early stages of oxidation prevents development of the cation-deficient FeO layer normally found on iron at temperatures above 570°C. The oxidation is thus controlled by the migration of iron and oxygen through the more diffusion-resistant Fe_3O_4 and Fe_2O_3 layers.

Addition of molybdenum has a much less potent effect on the oxidation of nickel. In amounts up to 13·1 at. per cent. molybdenum the oxidation rate of the alloys is slightly increased by comparison with that of nickel, due to formation of additional vacancies in the NiO lattice brought about by dissolved molybdenum atoms. Above 13·1 at. per cent. molybdenum, however, the MoO_2 subscale becomes sufficiently dense to hinder migration of nickel ions, and the oxidation rate falls. It is postulated that the controlling step in the oxidation process continues to be migration of nickel ions across the NiO layer. This assumption is confirmed by the small changes observed in the rates of oxidation and heats of activation of the alloys.

See also—

S. S. BRENNER: 'Catastrophic Oxidation of Some Molybdenum-containing Alloys.' *ibid.*, pp. 16–21.

Study of the influence of nickel and chromium additions, to molybdenum-iron alloys. The alloys exposed (two hours at 1000°C.) are shown in the following table.

Mo %	Fe %	Ni %	Mo %	Fe %	Ni %	Cr %
20	80		10	85		5
20	75	5				
20	70	10	5	80		15
20	65	15	15	75		10
20	60	20		75		25
20	55	25	20	75		5
20	50	30	15	70		15
20	40	40	20	70		10
20	20	60	5	70		25
			10	65		25
	75	25	30	65		5
5	70	25				
10	65	25	*20	35	40	5
30	45	25	*20	30	40	10
30	20	50	*20	20	40	20

The data recorded for the respective alloys show that when nickel is substituted for iron in the 20–80 molybdenum–iron alloy there is, up to about 10 per cent. of nickel, a slight decrease in rate of oxidation. From about 10 per cent. of nickel, however, there is an increase, which becomes rapid, with a peak at 25 per cent. nickel, where the oxidation rate is about fifteen times as fast as that shown by an alloy containing molybdenum 20, iron 70, nickel 10, per cent. Beyond 25 per cent. nickel the oxidation rate of the binary alloys slows down very markedly, and at 40 per cent. of nickel approaches that of the 20–70–10 alloy molybdenum–iron–nickel. Further increase in nickel, up to 60 per cent., results in only slight change in rate of oxidation.

Additions of chromium to iron–molybdenum alloys initiate rapid oxidation even more quickly: small amounts of chromium (about 5 per cent.) are sufficient to accelerate attack. The effect of additions of more than 25 per cent. chromium could not be evaluated, due to difficulty in fabricating the alloys.

It is noted that when the alloys contain a sufficiently high percentage of nickel, formation of MoO_3 is prevented (cf. the nickel-rich nickel–molybdenum alloys). The three alloys containing 40 per cent. of nickel (starred in the table) showed no sign of catastrophic oxidation, and it is suggested that these compositions may be industrially useful.

With regard to the mechanism of catastrophic oxidation, it is suggested that immediately after the initial oxidation of the alloy the metal surface becomes enriched in molybdenum. The oxide coating cracks, allowing the formation of liquid MoO_3 along the alloy/oxide interface. MoO_3 constantly oxidizes the iron and chromium in the alloy to Fe_2O_3 and Cr_2O_3, which dissolve in the MoO_3 and re-precipitate along the MoO_3/bulk oxide interface, preventing healing of the cracks.

Corrosion of High-Temperature Materials by Vanadium Pentoxide

S. H. FREDERICK and T. F. EDEN: 'Corrosion Aspects of the Vanadium Problem in Gas Turbines.' *Corrosion*, 1955, vol. 11, Jan., pp. 35–45; disc., pp. 45–9.

Reprint from *Proc. Instn. Mechanical Engineers*, 1954, vol. 168, Mar., pp. 125–34. (For abstract of paper see *Nickel Bulletin*, 1953, vol. 26, No. 12, pp. 212–13.)

A. DE S. BRASUNAS: 'Catastrophic Corrosion Resulting from Vanadium Pentoxide in Fuel-Oil Ash.' *ibid.*, 1955, vol. 11, Jan., pp. 17–18.

This note, presented as 'Topic for the Month', describes the character of the accelerated corrosion resulting from small amounts of vanadium pentoxide present in the ash derived from fuel oils. Theories which have been evolved to explain the attack are discussed and illustrations show typical deterioration which may arise from this cause. Sources of vanadium-bearing oils are discussed, and the amounts present in various types of oil (and in the resulting ash) are listed. *Inter alia*, the author illustrates the molecular structure of vanadium porphyrin, an oil-soluble compound, which is the cause of the unusual corrosion. Attention is directed (with bibliographical references) to some of the major researches on this problem. To date, the conclusions are that nickel-rich alloys are somewhat more resistant than nickel-free alloys to this form of corrosion attack, and the use of deterrent additives in the oils also offers some promise, but further research is needed before a practical large-scale solution is in sight.

Palladium-Nickel-Chromium Alloy for Brazing High-Temperature Alloys

H. A. SALLER, J. T. STACEY and H. L. KLEBANOW: 'High-Temperature Brazing of Nichrome V.' *U.S. Atomic Energy Commission Report* BMI-933, Aug. 2, 1954; 26 pp.

For many purposes the 80–20 nickel–chromium alloy which is so generally used for electrical-resistance heating can be brazed with an alloy of the Nicrobraz type (nickel–chromium alloy containing boron). The presence of boron in the brazed joints is, however, unacceptable for certain applications in nuclear plant, and, to obviate difficulties arising from this source, a boron-free brazing alloy has been developed by the General Electric Company, U.S.A. This material, having the designation G.E. 76, contains palladium 44, nickel 33, chromium 23, per cent.

The work reported was done to provide design data for use of the modified alloy. Tensile strength and ductility values of brazed joints were determined at room temperature, and at 1200°, 1650° and 1800°F. (650°, 900° and 980°C.), and similar tests were made, at the same temperatures, on the brazing alloy itself. Since particular interest attaches to the use of thin nickel–chromium alloy sheet, the experiments were made with sheet 0·015 in. thick.

The palladium–nickel–chromium alloy is reported to show excellent wetting characteristics on 80–20 nickel–chromium alloy at 2320°F. (1270°C.) in dry hydrogen. It is recommended that to obtain heavy braze fillets under these conditions the clearances of the nickel-alloy joints should be kept to a minimum.

The brazing alloy is hot-short at 1200°F. (650°C.) and above, a characteristic which is believed to be attributable to the presence of impurities such as sulphur and silicon. As a result, T joints in the 80–20 alloy brazed with the palladium-base material show a rapid drop in ductility at 1200°F. (650°C.) and above. Annealing of the brazed joints severely reduces room-temperature ductility, but slightly raises ductility at high temperatures. At room temperature and at 1200°F. (650°C.) the 80–20 nickel–chromium alloy is stronger than the ternary brazing alloy, but at 1650° and 1800°F. (900° and 980°C.) the reverse is the case. Butt joints brazed with the new alloy are intermediate in strength between the parent material and the brazing alloy.

The two materials are compatible in thermal-expansion coefficients over the range of temperature used in these experiments. At room temperature the modulus of elasticity of the brazing alloy is about 60 per cent. that of the 80–20 nickel–chromium alloy.

U.S. Government Specifications for Stainless Steels

'Government Specification Chart for Stainless Steel.' *Materials and Methods*, 1954, vol. 40, Dec., pp. 131–3.

Tabular summary of compositional requirements laid down for stainless steels covered by the following groups of U.S. Government specifications: Federal (QQ series), Military (MIL and JAN series), Army-Navy Aeronautical (AN series), Navy (numbered) and Army (numbered).

The A.I.S.I. types to which the respective steels conform are also indicated.

Influence of Heat-Treatment on Fatigue Strength of Nickel-Chromium Steels

I. A. ODING and A. P. SHISHKOVA: 'Influence of Preliminary Treatment on the Fatigue Strength of Austenitic Steel.' *Vestnik Mashinostroeniya*, 1954, vol. 30, No. 7, pp. 40–5 ; *Chemical Abstracts*, 1954, vol. 48, pp. 12006–7.

Report of tests on steels of the following compositions:—

C %	Cr %	Ni %	W %	Mo %
0·13	14·5	13·95	2·14	0·5
0·47	14·1	13·85	2·55	0·3

The steels were tested in the following conditions:—

Quenched from 1180°C.

Quenched from 1180°C. and aged at 400°–450°C. for 400 hours.

Quenched from 1180°C. and aged at 600°C. for 75–150 hours.

Quenched from 1180°C. and aged at 750°C. for 25–75 hours.

Test conditions: 10×10^6 cycles in a cantilever-beam machine, at 20°–700°C.

In the quenched condition, the fatigue strength of the steels containing 0·13 per cent. of carbon fell with rise in test temperature; un-notched test pieces of the 0·47 per cent. carbon type became stronger as the test temperature was raised, but the reverse effect was found with notched specimens. Ageing was without effect on the fatigue strength of the lower-carbon steel; in the 0·47 per cent. carbon steel the fatigue strength of smooth specimens fell with rising temperature, but that of the notched specimens was increased. In both steels quenching and cold-working caused a rise in mechanical strength and reduction in ductility.

Influence of Heat-Treatment on Cold-Worked Austenitic Steel Wire

S. STORCHHEIM: 'The Response to Heat-Treatment of 18–8 Wires Given Various Per Cent. Cold Reductions.' *Wire and Wire Products*, 1954, vol. 29, Nov., pp. 1327–30.

This paper reports a section of the work done in the laboratories of the Wilbur B. Driver Company, to investigate the properties which can be developed, by cold-work and heat-treatment, in 18–8 stainless steel of the type used for magnetic recording. Earlier papers in the series have covered (*a*) the response of this steel to cold-work, and (*b*) the response, to heat-treatment, of wires of 18–8 steel which had undergone given percentages of cold reduction, to a magnetic condition. The present paper, which is an extension of that work, deals with the response, to heat-treatment, of wires of two types of steel (see table below) which had undergone varying percentages of cold reduction.

Initial preparation of three sets of each wire consisted in cold reduction, and a low-temperature annealing treatment (at 1225°F.: 663°C.). The diameters of the wires at this stage were such that further drawing to a diameter of 0·004 in. involved reductions of 80, 94 and 98 per cent., respectively. At the 0·004-in. stage each of the wires was heat-treated at temperatures within the range 635°–670°C., at intervals of 5°C. Full details of the results are shown in tables and curves.

The main deductions are as follows:—

In all cases there is a fall in remanence as the temperature of treatment is raised, and the indication is that the greater the percentage of previous cold reduction the more drastic is the fall in remanence resulting from increase in the temperature of treatment. Rise in temperature of heat-treatment causes increase in coercive force, and the rise takes place more rapidly in the more severely cold-worked wires.

The relation between structure, composition and response to heat-treatment is discussed, and it is noted that in the case of the 80 per cent. reduced wire curves for both remanence and coercive force *vs.* temperature of treatment show relatively flat portions, indicating that material which has undergone that degree of cold work is less susceptible than more severely worked wire to conversion of martensite to austenite by heat-treatment. This fact suggests that in production operations, a wider range of heat-treatment temperature would be permissible with wire in that condition than could be allowed for wires which had been more drastically reduced.

Steels Investigated

Type of Steel	Ni %	Cr %	Mn %	Si %	C %	Mo %	Fe %
A.I.S.I. 302	9·01	18·62	0·89	0·42	0·100	—	Bal.
A.I.S.I. 304	9·11	18·77	0·56	0·55	0·065	0·34	Bal.

Cold-Heading of Stainless Steel

W. M. BALDWIN and C. A. BEISER: 'Why Stainless is Hard to Cold-Head.' *Iron Age*, 1955, vol. 175, Jan. 13, pp. 82-5.

The fact that stainless steel does not head-up so readily as plain carbon steel has presented problems to bolt makers and others who use stainless types in high-speed heading operations. This article reports tests made to determine the cause of the embrittlement liable to occur in cold-heading of austenitic steels and to establish conditions in which cold heading can be carried out without impairing the quality of the bolts. Comparative compression tests, carried out on carbon and stainless steels over a wide range of forming speeds, showed that whereas, at all the speeds used, carbon steel can be cold-headed without difficulty to about twice its diameter, stainless steel can be successfully headed only at slow speed. If high speeds are used embrittlement occurs and full heading cannot be achieved: when deformed at very high speeds the steel actually behaves in a glass-brittle manner. (Although the decrease of ductility with speed of deformation in austenitic steels had previously been established, the extent of embrittlement produced by the very fast rates of forming has not been fully realized. Most of the ductility data which had been obtained on stainless steel had been derived from tensile tests, in which sensitivity to rate of deformation is not so high as in cold-heading.)

In an attempt to arrive at commercially practicable cold-heading conditions which would obviate embrittlement in stainless steel bolts, various factors were studied:—Slight modification in ratio of free length to diameter produced some improvement, but not to a degree warranting further consideration, and the use of indented dies also effected only minor improvement. Heading over a range of temperatures (−321° to +400°F.; −196° to +205°C.) indicated that rise of temperature was to some extent beneficial. Because many heading operations comprise two or more strikes, the effects of two or of three blows, on ultimate ductility, was examined, and the influence of the time interval between the blows was also considered. The results led to the conclusion that although substantial improvement is possible by a two-blow procedure, the time between the blows is so highly critical that a two-strike method is not a practical proposition. A three-blow design, with four minutes between the strikes, was also found to effect some improvement in the cold-heading limits.

From the above tests it emerged that but little could be done by mechanical modification of the cold-heading operation, and consideration was therefore given to selection of steel compositions which would permit reasonable speeds of heading. Tests were made on a range of iron-nickel-chromium alloys, on pure nickel, and on some ferritic steels: cold-heading limits were determined at various speeds of deformation. High-speed embrittlement was observed in a wide range of the austenitic steels, but was not observed in pure nickel or in the ferritic steels examined. Embrittling of the austenitic steels was not confined to those which undergo austenite-to-martensite transformation during deformation, and the authors note that further studies, of which the results are not yet published, confirm that the austenite-martensite transformation appears to be in no way associated with high-speed embrittlement.

Influence of Design and Fabrication on Performance of Stainless-Steel Equipment

J. A. COLLINS: 'Effect of Design, Fabrication and Installation on the Performance of Stainless-Steel Equipment.' *Corrosion*, 1955, vol. 11, Jan., pp. 27-34; disc., p. 34.

The increasing use of stainless steels in chemical equipment has served to demonstrate the extent to which design can support the full exploitation of the intrinsically valuable properties of these materials, and, conversely, has revealed the possibility of unsatisfactory behaviour due not to the material, but to mechanical factors. The author of this paper gives details of some case histories of failure in molybdenum-containing and niobium-stabilized 18-8 chromium-nickel steels, in which faulty behaviour has been traceable to unsuitable design, fabrication, or installation procedure.

Following some general and well illustrated recommendations with regard to design which will favour prolonged life, details are given of failures of the following types:—

General and crevice-cell corrosion in vertical tube condensers; thermal-corrosion failure in the tube sheet and tube ends of a waste-heat boiler; leaks in welded steel tubing handling hot wash water; corrosion failure of a wash compartment handling dilute sulphuric acid, hydrogen sulphide and carbon disulphide, at ambient temperatures. The nature of the failures is described in some detail, the failed parts are illustrated, and particulars are given of remedial measures which have prevented recurrence of breakdown.

Influence of Minor Elements on Intergranular Corrosion in Austenitic Steels

J. J. HEGER and J. L. HAMILTON: 'Effect of Minor Constituents on the Intergranular Corrosion of Austenitic Stainless Steels.' *Corrosion*, 1955, vol. 11, Jan., pp. 22-6.

Intergranular corrosion of the austenitic stainless steels is generally believed to be due to grain-boundary precipitation of chromium carbides, resulting in depletion of these zones in chromium and lessened resistance to corrosive attack. To overcome this susceptibility, steels having a carbon content of not more than 0·03 per cent. have been developed, and in many conditions such steels have shown immunity to this type of deterioration. It has, however, been observed that if extra low-carbon steels are heated for very long periods in the sensitizing range of temperature they show susceptibility in the standard boiling 65 per cent. nitric-acid test, even though the

carbon content may be as low as 0·017 per cent. (Typical data are shown to illustrate this observation.) This finding, which suggested that formation of chromium carbides is not alone responsible for intergranular corrosion, has been confirmed by results of tests on 18-8 chromium-nickel steels containing molybdenum. Sensitized 18-8-Mo steel containing 0·03 per cent. of carbon corroded, in the nitric-acid test, at about the same rates as steels of the same type but containing higher carbon. In addition, it was found that additions of niobium to these steels brought about only slight reduction in the rate of corrosion, and that the niobium-stabilized extra-low-carbon 18-8-Mo corroded more rapidly than the niobium-stabilized steels of higher carbon content. It has been suggested that sigma phase may be a 'minor constituent' partially responsible for the susceptibility of the 18-8-Mo steels, but the present authors consider that this explanation should not be accepted unless and until it is supported by further evidence of a more conclusive nature than any which has yet been advanced.

In the present work metallographic studies were made on the 18-8-Mo steels, using the nitric-acid corroding solution as etchant, and the results revealed that the delta-ferrite/austenite grain boundaries were severely attacked, with complete loss of some of the delta-ferrite grains. It is therefore postulated that during sensitization some minor constituent has formed at the grain boundaries of the low-carbon steels and that the rate of formation of this constituent was extra rapid at the boundaries separating the delta ferrite from the austenite. Thus a steel containing delta ferrite will exhibit high corrosion rates in the boiling-nitric-acid test, because the delta-ferrite grains actually drop out. When the delta-ferrite content is held to a minimum lower rates of corrosion will be found.

Summarizing, it is conceded that the principal minor constituent influencing susceptibility to intergranular corrosion is chromium carbide, but it is believed that other minor constituents, not yet positively identified, also play a part.

Corrosion in the Chemical and Petro-Chemical Industries: U.S. Experience

ORGANIZATION FOR EUROPEAN ECONOMIC CO-OPERATION 'Corrosion Problems and Prevention in the Chemical and Petro-Chemical Industries in the U.S.A.'
Report of Technical Assistance Mission No. 130.
Published by O.E.E.C., Paris, Aug., 1954; 129 pp.

The report was prepared by a Mission sent by the Chemical Products Committee of O.E.E.C. (in 1953) to the U.S.A., to study problems of maintenance in the chemical industry, with special reference to corrosion. The Mission was divided into three parties, charged, respectively, with investigating experience and practices in the heavy-chemical, the light-chemical, and the petro-chemical industries. The main task of the Mission was the study of methods and materials used in U.S.A. but less familiar in Europe.

The information given in the report is based on visits made to nearly forty research institutes and industrial firms, and on many informal contacts with individuals. Presentation is under three main headings:—

I. *Theory and Method*, including discussion of the economics of corrosion research which has been carried out in the U.S.A.; the importance of design in prevention of corrosion; methods of corrosion testing in current use; the rôle of the corrosion engineer, and the work of the National Association of Corrosion Engineers.

II. *Protective Techniques*, including the use of 'protective barriers' of paint or other non-metallic substances, the use of cathodic protection, and methods especially applicable in the petroleum industry, e.g., use of inhibitors, treatment of waters, etc.

III. *Anti-Corrosive and Heat-Resisting Materials*

(a) *Metallic Materials*, including reference to experience with, and current practice in the use of, unalloyed steels, medium- and high-alloy steels and cast irons, and special low-iron or iron-free alloys, e.g., Inconel, nickel-molybdenum-base alloys of the Hastelloy and Chlorimet types, cobalt-base (Stellite-type) alloys, nickel-chromium-boron facing alloys (Colmonoy), Monel, copper-nickel and other copper-base alloys, aluminium-base alloys, titanium, tantalum, and molybdenum.

(b) *Non-Metallic Materials and Cermets*, including glass and ceramics, cements, and plastics.

On the basis of the information obtained, some recommendations are made with regard to increasing the effectiveness of the fight against corrosion, which is already being actively pursued in many European countries.

Corrosion of Condenser Tubes in contact with Polluted Waters

J. CHANGARNIER: 'Corrosion of Condenser Tubes Handling Harbour Waters.'
Corrosion et Anti-Corrosion, 1953, vol. 1, Mar.-April, pp. 8-9.

The author reports a case of rapid and severe deterioration of 70-30 cupro-nickel condenser tubes in a power station: the tubes were working with polluted water in a dock area. The attack was due to combined corrosion and erosion, which prevented formation of, or destroyed, the protective film which would normally be present on tubes of this alloy in sea-water service. A condition of passivity was successfully induced by subjecting the tubes, after cleaning, to treatment in a permanganate solution, and the tubes so treated are giving satisfactory service.

See also—

A. J. MAURIN: 'Corrosion Phenomena Observed on Condenser Tubes, and Remedial Measures.' *ibid.*, May-June, pp. 56-66; disc., pp. 67-9.

In this lecture corrosion attack involved in condenser-tube service is considered in greater detail and from a more fundamental angle. The service experience in the instance recorded above is discussed,

and an illustrated survey is made of the types of corrosion occurring in condenser tubes. Materials suitable for service in specific conditions are reviewed, and it is urged that there is a need for some revision and amplification of specifications governing supply of condenser tubes. Suggestions are made as to requirements which should be embodied in such specifications. The phenomenon of cavitation, arising particularly in marine propellers, is also discussed.

Nickel Alloys for Handling Aqueous Hydrofluoric Acid

M. SCHUSSLER: 'Metal Materials for Handling Aqueous Hydrofluoric Acid.'
Industrial and Engineering Chemistry, 1955, vol. 47, Jan., pp. 133–9.

Although many materials have useful resistance to aqueous hydrogen fluoride when completely immersed in the liquid, severe attack may in some cases occur at the liquid-level line or at points where condensation of vapour occurs. This aspect is currently under investigation.

The present paper is concerned with research prompted by the observation that leaks frequently develop in joints of plant handling such solutions: specifically it deals with the corrosion-resistance of welded and silver-brazed joints in Monel and copper-nickel, brazed joints in Illium R*, and unwelded lead and silver, exposed to aqueous hydrofluoric acid at concentrations of 48 and 5 per cent. at 150°F.±10°F. (65°C.±5°C.) for about 30 days, in conditions representative of those encountered in service.

The rate and type of attack were determined by measurements of weight-loss, and by optical examination of the corroded specimens; the nature of the corrosion products was determined by chemical analysis. Tables and typical photographs and photomicrographs illustrate the results.

The conclusions drawn are summarized below:—

'Monel and copper-nickel showed little attack when completely immersed in the aqueous hydrofluoric acid baths. Either welded or silver-brazed joints in these materials should give good service under these ideal conditions. Furthermore, the welded joints could be used in the as-welded condition and, if silver-brazed joints are used, the selection of brazing alloy is not critical.

'The evidence obtained in this study indicates that the life of Monel and copper-nickel in hydrofluoric-acid service will be considerably decreased when a liquid-level line is involved, or when alternate evaporation-condensation of vapour containing oxygen occurs. The susceptibility of these materials to severe attack under these conditions is the most important consideration in their service life. Further, Monel (and perhaps copper-nickel also) can suffer stress-corrosion cracking under these conditions, particularly in welded areas. Silver-brazed joints of Monel show undermining of the brazing alloy and can show stress-corrosion cracking under these conditions if the Monel has sheared edges.

'These severe types of attack on Monel or copper-nickel may be avoided by keeping the metal surface continuously immersed in the liquid (by keeping the system full at all times), *or* purging the vapour with purified (oxygen-free) nitrogen, *or* maintaining the pressure in the system above atmospheric pressure, so that oxygen is excluded. If any or all of these conditions can be incorporated in the design, either Monel or copper-nickel should prove to be a suitable long-life material of construction when the process conditions involve acid concentrations and temperatures similar to those studied in this investigation.

'Some acid-handling systems cannot, however, be kept full of liquid, and it is usually impracticable completely to exclude oxygen. It is considered doubtful whether any industrial hydrofluoric-acid application can be regarded as involving the ideal exposure conditions of a quiet bath and total immersion, and it therefore appears necessary to incorporate certain precautionary measures when using Monel in such systems. It is recommended that only fully annealed Monel be used for fabrication, and that the welding procedure should be such as to avoid ragged projections or crevices. The shielded inert-gas process is recommended for welding, and the weld areas should be stress-relieved, or, preferably, given a full annealing treatment, followed by slow cooling. Silver-brazed joints should have long lap lengths (suggested as a minimum of three to five times the base metal thickness) and should rigidly conform to proper joint clearances in order to obtain high strength and freedom from crevices. Only brazing alloys of high silver content should be used, and sheared or severely cold-worked edges on the base metal should be avoided. It would also be safe practice to apply the above precautions to systems fabricated from copper-nickel.

'When alternate condensation-evaporation of aqueous hydrofluoric-acid vapours containing oxygen cannot be avoided, the system should be designed in such a way as to ensure that these severe conditions are confined to localized areas. Among the materials tested, only silver offered superior resistance to attack at and above the liquid-level line. In this type of service pure silver or, if the temperature is low enough, certain plastics or impervious graphite may offer the lowest-cost materials of construction, considering their long expected service life.

'Neither Illium nor lead appeared to have useful resistance to corrosion by aqueous hydrofluoric acid under the test conditions. Even when totally immersed, Illium R was perforated under some test conditions. Lead did not appear to resist the general chemical attack.'

Properties of the Heat-Affected Zones in Welded Niobium-Stabilized Austenitic Steel

E. F. NIPPES, W. WAWROUSEK and W. L. FLEISCHMANN: 'Some Properties of the Heat-Affected Zone in Arc-Welded Type 347 Stainless Steel.'
Amer. Soc. Mechanical Engineers, Paper 54-A-57, Nov.-Dec., 1954; 14 pp. + figures and tables.

The paper describes an investigation carried out in

* Typical composition: Nickel 58, chromium 21–23, copper 0–4, molybdenum 4–6, per cent. balance iron

connexion with the occurrence of 'knife-line' attack in the heat-affected zone of welded niobium-stabilized 18-8 chromium-nickel steel. Section I of the report covers a study of the thermal cycles occurring at various locations in the weld-heat-affected zone of 1½-in. thick stainless-steel plate; Section II gives test results obtained on specimens of material which had undergone cycles typical of those producing the characteristic heat-affected zone observed in the welded steel. The data include microstructures, details of impact strength at 1100°F. (593°C.), and observations on the effect of a sensitization treatment on resistance to attack in the boiling 65 per cent. nitric-acid (Huey) intergranular-corrosion test.

Microstructural changes, followed up to a region which had attained a peak temperature of 2500°F. (1370°C.) showed that grain-coarsening and incipient fusion occurred with temperatures of 2475° and 2500°F. (1356° and 1370°C.). In the heat-affected zone two distinct niobium-bearing phases were observed: these reacted differently during the welding cycle. 'Dot' carbides went into solution at temperatures below 2475°F. (1356°C.), whereas the other phase remained at the grain boundaries, and showed definite signs of melting at 2500°F. (1370°C.). In the boiling nitric-acid test 'knife-line' attack occurred only in regions which had been heated to temperatures above 2400°F. (1315°C.). Room-temperature impact strength of the heat-affected zone, in the as-welded condition, was found to be not appreciably different from that of the base metal. The rupture strength of the region which had attained the 2400°F. (1315°C.) peak temperature showed considerable loss in ductility by comparison with the parent material.

Determination of Nickel and Other Elements in High-Temperature Alloys

J. L. HAGUE, E. E. MACZKOWSKE and H. A. BRIGHT: 'Determination of Nickel, Manganese, Cobalt, and Iron in High-Temperature Alloys, using Anion-Exchange Separations.' *Jnl. of Research, Nat. Bur. Standards*, 1954, vol. 53, Dec., pp. 353-9: R.P. 2552.

Much attention has recently been directed, in the literature of analytical chemistry, to difficulties arising in the estimation of cobalt, iron, and nickel when present in combination; the problem has become increasingly urgent in connexion with the development of both high-temperature alloys and 'special-property' alloys, e.g., those having closely controlled expansion characteristics, or magnetic properties of a particular order. The difficulty is enhanced by the presence, in many of these alloys, of small amounts of other elements.

In the present paper, which describes the application of anion-exchange separations to the analysis of high-temperature alloys, the following procedure is recommended:—

Niobium, tantalum, tungsten and silicon are rendered insoluble by hydrochloric-acid dehydration, after solution of the alloy in aqua regia. Molybdenum and copper are precipitated with hydrogen sulphide, and the solution is filtered. The filtrate is evaporated several times with hydrochloric acid and hydrogen peroxide, to obtain a chloride solution of these elements in the correct valence, and the resulting solution is transferred to an anion-exchange column containing Dowex 1. Nickel and manganese, with chromium and some other elements, are collected in the first fraction, by elution with 9-*N* hydrochloric acid. Cobalt is collected in a second fraction by elution with 4-*N* hydrochloric acid, and iron in a third fraction, by elution with 1-*N* hydrochloric acid. Cobalt is determined by electrolysis from a weakly ammoniacal chloride solution containing hydroxylamine hydrochloride, and the other elements are estimated by conventional methods. Full details of procedure are given.

Results obtained by these methods, on a series of synthetic solutions made to simulate typical nickel- and cobalt-base alloys, are quoted in the paper.

Determination of Titanium in Steel

J. R. SIMMLER, K. H. ROBERTS and S. M. TUTHILL: 'Separation of Titanium combined with Spectrophotometric Determination of Titanium in Steel.' *Analytical Chemistry*, 1954, vol. 26, Dec., pp. 1902-4.

In developing a specific qualitative test for titanium, FEIGL and RAJMANN showed that titanium is co-precipitated with zirconium arsenate from a strongly acid solution; they used this effect as a means of separating small amounts of titanium from large amounts of iron, vanadium, and chromium (*Mikrochemie*, 1935, vol. 19, pp. 60-3). Such procedure suggested the possibility of quantitative determination of titanium in steels by combining this separation with a spectrophotometric method for the final estimation. The present paper describes a method based on such combined procedure.

Confirmatory analyses quoted in the paper include determinations of titanium in Bureau of Standards standardized stainless steels of nickel-chromium types.

Chemical Analysis of Steel: German Practice

See abstract on p. 47.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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